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Ion Exchange Studies of Synthetic (Al, Ge) Gismondine

A. Celestian, J. Parise (SUNY, Stony Brook), and J. Hanson (BNL)

Beamline(s): X7B

Introduction: The (Al, Ge) Gismondine (GIS) analogue to the natural (Al, Si) GIS has been under scrutiny ever since the structure was solved by Tripathi et. al. [1]. This study examines the two end members Na-GIS (ordered super-cell) and K-GIS (disordered sub-cell) where the Na and K are the extra framework cations in the channels of the nano-porous material. The reason for the interest in these two end members is the volume and internal symmetry change seen from single crystal diffraction. Na-GIS has a much larger unit cell volume (approx. 3 times larger than the K-GIS counterpart) and studying the solid solution between the two end members will give insight to how this change occurs.

Methods and Materials: The experiment started with Na-GIS mounted into a .7 mm quartz capillary that is fitted into an apparatus for *in situ* time resolved ion exchange. A .03 M solution of KCl will pass through the sample as diffracted x-rays are collected on an imaging plate. The drop rate of the solution through the sample was calibrated to be 1 drop/min. and continued at this pace for 6 hours. The experiment was conducted at room temperature.

Results: At the end of the run, it was seen that the larger Na-GIS unit cell did change to a much small unit cell at 50% exchange K for Na and 100% exchange at the end of the six hour run.

Conclusions: Until the structure refinement is done, the extra framework atomic positions cannot be determined. What can be determined is unit cell parameters and how they change through time of ion exchange. As seen in figure 1, the two quadruplets at the beginning of the experiment are reduced to a triplets at the end of the first cluster of diffraction patterns. This shows the change from the super-cell to the sub-cell at 50% exchange K for Na. At the end of the second cluster, the first triplet remains a triplet while the second triplet becomes a doublet. This denotes when the material is nearly 100% K for Na exchanged.

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Reference:

[1] A. Tripathi, J.B. Parise, S.J. Kim, Y. Lee, G.M. Johnson, and Y.S. Uh "Structural Changes and Cation Site Ordering in Na and K Forms of Aluminogermanates with the Zeolite Gismondine Topology" Chem. Mater. 2000, 12, 3760-3769.

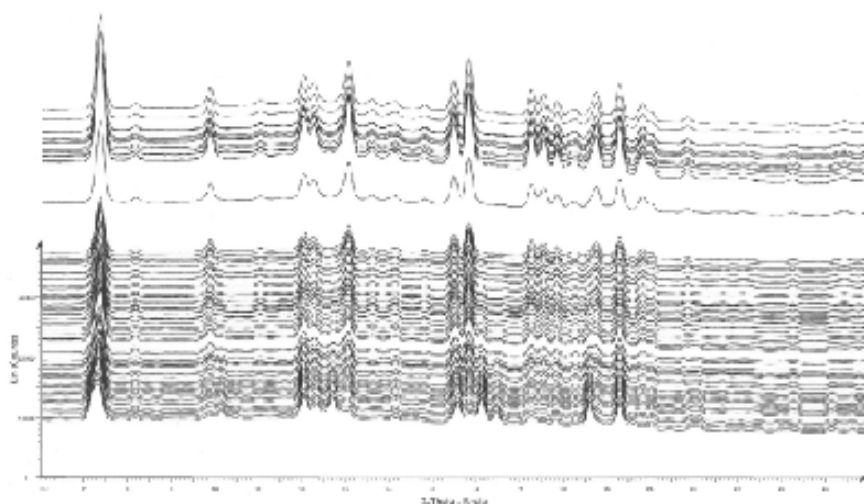


figure 1. *In situ* ion exchange of Na-GIS with KCl